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Carbon Nanotube Sensors and Field Emitters

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Final Report for PHYS 4900

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The first project that I pursued for PHYS 4900 was using carbon nanotube forests as biological sensors, which was partially funded by an URCO grant. However, after completing the first part of the project it was realized that the second part would not function as expected. This semester I have been attempting to use bundles of carbon nanotubes as field emitters. Both projects will be described in this report.

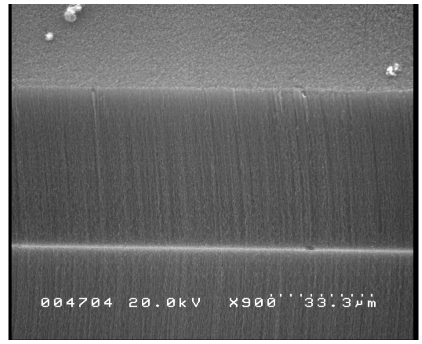

Fig. 1a. A pristine CNT forest, seen from a 45-degree angle from the top.

Functionalizing Carbon Nanotube Forests

Carbon nanotube (CNT) forests are arrays of free-standing CNTs, as seen in Fig. 1a. The goal of this project was to deposit 1,5-diaminonaphthalene (DAN) evenly on each CNT. The motivation is that CNTs cannot effectively participate in chemical reactions by themselves. However, DAN can bind to the CNT surface in such a way that it can participate in chemical reactions while staying on the CNT side wall[1]. If DAN could be coated evenly on the CNTs throughout the forest, it could make a very sensitive biological sensor. A sensor is only as good as the number of detection sites, and the large surface area in a CNT forest provides ample opportunity for many detection sites. Initially, we tried immersing CNT forests (of about 50 μm height) in solutions of DAN, but it was found that the CNT forest deformed so much that there was little CNT surface to which DAN could bind. The result is seen in Fig. 1b, and no DAN was detected via fluorescence spectroscopy.

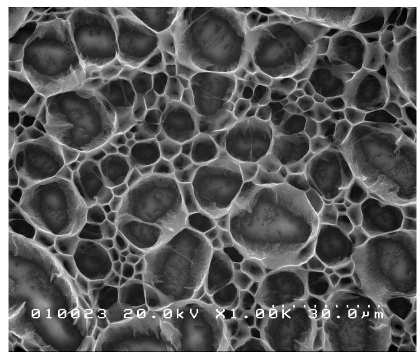

Fig. 2b. A CNT forest deformed by wetting, seen from directly overhead.

Part of the project was to try a chemical vapor deposition (CVD) technique to deposit DAN evenly throughout the CNT forest in the vapor phase, and provide favorable conditions for condensation. A diagram of the high-vacuum CVD chamber that was constructed is found in Fig. 2.

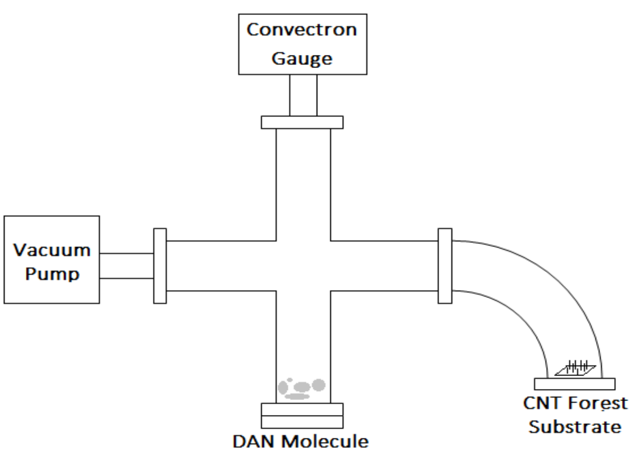

Fig. 3. The DAN molecule was put in the bottom of the chamber, where there was a window to see if it had evaporated. The vacuum pump was used to reduce the pressure to $4*10^{-2}$ torr (as measured by the convectron gauge), at which point the chamber was closed. The end with DAN was placed in an oil bath for various exposure times at 250 °C. The high temperature was needed to evaporate the molecule.

In every test, DAN crystals formed on top and within the CNT forest, similar to the crystal shown in Fig. 3. Once crystals form, forming larger crystals is more thermodynamically favorable than making the thin film that we desired. Despite varying temperatures in the chamber and of the substrate, and the times of exposure, crystals always formed. Since the crystals are small and sparse, they could not be used in this application. In a preliminary study we found some planar crystals, and after further investigation we found that crystals only formed in the planar configuration, and deformed in a similar fashion to wetted CNTs of Fig. 1b.

Fig. 3. Formation of a large crystal on top of CNT forest

It was found that shorter CNT forests (about 25 μm height) did not deform when place in a liquid, and so a sample was placed in a highly concentrated solution of DAN to promote its dispersion throughout the forest. Crystals formed this time as well, but they appear to be spherical in shape, not planar. Though there is something interesting to be said about the different crystal shapes, the crystals are not useful for the goals of this study, for the same reasons listed above.

Electron Field Emission with Sharpened Carbon Nanotube Bundles

Electron beams are used in many applications, such as mass spectrometers, lithography, and testing applications. One way to make an electron beam is to apply a voltage to a very sharp metallic object. The voltage causes electrons to "jump out" of the sharp tip and accelerate away; this is called cold field emission. CNTs have been used in cold field emission, but a good balance between a robust CNT structure and satisfactory electron beam current has not been reached. Since using CNTs as a biological sensor platform had hit several large roadblocks, I decided to change research projects. The deformation seen in the figures above could be used to make ultra sharp CNT needles, which could possible function as electron field emitters.

I hypothesize that controlling the deformation of the CNTs while they are wetted could yield structures that are both robust and capable of high electron currents. Research about CNT deformation similar to this has already been carried out[2,3]. We are still in the process of testing this hypothesis, as various circumstances have pushed back the timetable significantly.

The first step was to create a pattern (for a photomask) to create the device. The whole fabrication process turns out to be about twenty steps. I used the software L-Edit to create the necessary patterns for this device.

I then used the software SIMION to simulate the carbon nanotube field emitters. It showed that the design that we had created could indeed work, and that there are several parameters that could be optimized in case of poor performance. In particular, it was found that the sharpness of the carbon nanotubes was very important to the performance of the CNT cold field emitters.

I have written a new LabView program to more efficiently control the mass-flow controllers while growing CNTs, and I am also currently writing another LabView program to control other aspects of lithography in the cleanroom.

Conclusion: It is very difficult to make biological sensors by coating CNTs with sensing molecules because of deformation, but it may be possible to control that deformation with lithographic and microfabrication techniques to make robust CNT electron field emitters. While it has not been possible to test this hypothesis yet, we are preparing to fabricate the field emitters soon.

References:

[1] R. J. Chen, *et al.* J. Am. Chem. Soc. **123** 3838 (2001).

[2] D. N. Futaba, *et al.* Nature Materials **5**, 987 (2006).

[3] G. Zhong, *et al.* ACS Nano **6**, 2893 (2012).